

PCTWORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁶ : D21H 11/00, 13/00, 15/00, 17/00, 19/00, 21/00, 23/00, 25/00, 27/00, C08L 53/00, 61/04, 39/00, 31/04, 27/00	A1	(11) International Publication Number: WO 98/55692 (43) International Publication Date: 10 December 1998 (10.12.98)
(21) International Application Number: PCT/US98/05261 (22) International Filing Date: 18 March 1998 (18.03.98) (30) Priority Data: 08/870,780 6 June 1997 (06.06.97) US (71) Applicant: BETZDEARBORN INC. [US/US]; 4636 Somerton Road, P.O. Box 3002, Trevose, PA 19053-6783 (US). (72) Inventors: LING, Tien-Feng; 2563 Stonebridge Drive, Jack- sonville, FL 32223 (US). SCHOLZ, William, F.; 10135 East Desert Cove, Scottsdale, AZ 85260 (US). (74) Agents: VON NEIDA, Philip, H. et al.; BetzDearborn Inc., 4636 Somerton Road, Trevose, PA 19053-6783 (US).		(81) Designated States: AU, CA, JP, KR, NO, NZ, SG, European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE). Published <i>With international search report.</i>
(54) Title: METHODS AND COMPOSITIONS FOR TREATING STICKIES (57) Abstract The present invention provides for methods and compositions for removing stickies and pitch from papermaking fiber by first chemically treating the papermaking fiber with a composition of at least two of an oil-soluble or water-dispersible hydrophobic polymer, an oil soluble or water-dispersible surfactant having an HLB of about 1 to about 10, a tackifier, and an oil-soluble or water-dispersible solvent then mechanically separating the agglomerated stickies and pitch from the papermaking fiber.		

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AL	Albania	ES	Spain	LS	Lesotho	SI	Slovenia
AM	Armenia	FI	Finland	LT	Lithuania	SK	Slovakia
AT	Austria	FR	France	LU	Luxembourg	SN	Senegal
AU	Australia	GA	Gabon	LV	Latvia	SZ	Swaziland
AZ	Azerbaijan	GB	United Kingdom	MC	Monaco	TD	Chad
BA	Bosnia and Herzegovina	GE	Georgia	MD	Republic of Moldova	TG	Togo
BB	Barbados	GH	Ghana	MG	Madagascar	TJ	Tajikistan
BE	Belgium	GN	Guinea	MK	The former Yugoslav Republic of Macedonia	TM	Turkmenistan
BF	Burkina Faso	GR	Greece	ML	Mali	TR	Turkey
BG	Bulgaria	HU	Hungary	MN	Mongolia	TT	Trinidad and Tobago
BJ	Benin	IE	Ireland	MR	Mauritania	UA	Ukraine
BR	Brazil	IL	Israel	MW	Malawi	UG	Uganda
BY	Belarus	IS	Iceland	MX	Mexico	US	United States of America
CA	Canada	IT	Italy	NE	Niger	UZ	Uzbekistan
CF	Central African Republic	JP	Japan	NL	Netherlands	VN	Viet Nam
CG	Congo	KE	Kenya	NO	Norway	YU	Yugoslavia
CH	Switzerland	KG	Kyrgyzstan	NZ	New Zealand	ZW	Zimbabwe
CI	Côte d'Ivoire	KP	Democratic People's Republic of Korea	PL	Poland		
CM	Cameroon	KR	Republic of Korea	PT	Portugal		
CN	China	KZ	Kazakhstan	RO	Romania		
CU	Cuba	LC	Saint Lucia	RU	Russian Federation		
CZ	Czech Republic	LI	Liechtenstein	SD	Sudan		
DE	Germany	LK	Sri Lanka	SE	Sweden		
DK	Denmark	LR	Liberia	SG	Singapore		
EE	Estonia						

METHODS AND COMPOSITIONS FOR TREATING STICKIES

FIELD OF THE INVENTION

5

The present invention relates to methods and compositions for removing stickies and pitch from papermaking fiber by a chemically enhanced mechanical separation process.

10 BACKGROUND OF THE INVENTION

In recent years, there has been an increasing use of recycled fibers in the paper industry due to environmental concerns, government legislation, economic considerations, and the market demand for paper
15 containing recycled fiber. Since stickies contaminants are present in varying amounts in recycled paper, they often cause special problems at various stages of the papermaking process. These problems often include:

- 20
- 1) increased machine downtime due to breaks and required clean-up
 - 2) reduced product quality due to picking, pinholes, and poor appearance

- 3) increased replacement costs for wires and felts
- 4) reduced converting and/or printing process efficiency
- 5) limited levels of fiber substitution
- 6) repulping difficulty

5

Stickies are generally a diverse mixture of polymeric organic materials which can stick on wires, felts or other parts of paper machines, or show on the sheet as "dirt spots". The sources of stickies may be pressure-sensitive adhesives, hot melts, waxes, latexes, binders for
10 coatings, wet strength resins, or any of a multitude of additives that might be contained in recycled paper. The term "pitch" normally refers to deposits composed of organic compounds which are derived from natural wood extractives, their salts, coating binders, sizing agents, and defoaming chemicals existing in the pulp. Although there are some
15 discrete characteristics, there are common characteristics between stickies and pitch, such as hydrophobicity, low surface energy, deformability, tackiness, and the potential to cause problems with deposition, quality, and efficiency in the process.

20 For the increased mandatory use of recycled fiber in the pulp and paper industry, co-occurrence of stickies and pitch problems are often observed. The materials of this invention have shown their ability to remove stickies from fiber furnish via agglomeration mechanisms and mechanical separation processes. It is believed that these materials can also be
25 used for pitch agglomeration and pitch removal from the fiber furnish.

Several non-chemical approaches have been used in the paper industry to combat stickies. These approaches include furnish selection,

screening, cleaning, and thermal/mechanical dispersion such as dispergers or kneaders. However, each of these approaches has limitations. Furnish selection may reduce but not completely eliminate the stickies' contaminants in the system, while it will significantly increase the furnish cost. Furthermore, paper companies will also tend to utilize lower grade furnishes to reduce raw material costs. Subsequently, even more contaminants are introduced into the papermaking system.

Stickies cannot be completely removed with screening equipment since minimum slot size is limited to 150 microns for throughput reasons. Finer screen may also cause a high level fiber loss with contaminants removal. Reverse and forward cleaning may not be effective for removing stickies due to the small density differences between stickies and water. Thermal/mechanical dispersion units can enhance the breakup of stickies, but cannot prevent the agglomeration downstream where problems usually occur. In addition, this approach requires capital investment costs and energy expenditure.

Chemical treatment techniques for controlling stickies and pitch include dispersion/emulsification, detackification, wire passivation, and cationic fixation. Chemical treatments include mineral additives, alum, polyphosphates, emulsifiers, dispersants, water-soluble polymers, and combinations thereof.

Typically, these agents are fed to the pulper, stock storage chest and/or applied to the process equipment surfaces where depositions occurred. These surfaces include paper machine foils, paper machine wires, wet press felts, dryer felts, Uhle boxes and calendar stacks.

SUMMARY OF THE INVENTION

The present invention is directed towards stickies and pitch
5 removal by chemically enhanced mechanical separation processes. The
stickies and pitch contained in the papermaking fiber are first treated
chemically and removed subsequently through a mechanical separation
process.

10 The chemicals employed in the present invention are either oil
soluble or water dispersible. These chemicals include at least two of an
oil soluble or water-dispersible polymer, an oil soluble or water-dispersible
surfactant having an HLB of about 1 to about 10, a tackifier, and an oil
soluble or water-dispersible solvent.

15

DESCRIPTION OF THE RELATED ART

A number of chemicals have traditionally been used to treat
stickies. Representative examples include a methyl cellulose derivative,
20 a methyl ether cellulose derivative, polyvinyl alcohol and urea. M. R.
Doshi in "Additives to Combat Sticky Contaminants in Secondary Fibers",
Tappi Recycling Paper: From Fiber to Finished Product, p. 519, 1991,
discusses five different additives for controlling stickies. These additives
include talc, solvents and dispersants, synthetic fibers, zirconium
25 compounds and sequestering agents for excess alum.

"Cost-Effective, Common Sense Approach to Stickies Control", T.
J. Fogarty, Tappi J., Vol. 76, No. 3, March 1993, provides an overview

addressing control methods such as dispersion, detackification, cationic fixation, wire passivation, and solvents to control stickies in recycled fiber.

All of these approaches will allow the stickies to either attach to the fiber surfaces or remain in the papermaking water system. For the stickies adhered to the fiber surfaces, they will eventually contaminate the system when the fiber is recycled. Stickies remaining in the water system will cause problems later due to re-agglomeration, deposition, etc., especially for a closed loop system. In comparison, the present invention is able to make stickies agglomerates, which are not attached to the fiber surfaces, and completely remove them from the papermaking system.

DETAILED DESCRIPTION OF THE INVENTION

The present invention relates to methods and compositions for removing stickies and pitch from papermaking fiber comprising the steps of adding to the papermaking fiber in the pulper or stock storage chest a composition comprising at least two of an oil soluble or water-dispersible hydrophobic polymer, an oil soluble or water-dispersible surfactant having an HLB of about 1 to about 10, a tackifier, and an oil soluble or water-dispersible solvent, wherein the composition will cause the agglomeration of stickies and pitch, and a mechanical separation of the stickies and pitch from the papermaking fiber.

The present inventor has discovered that the inventive two-step approach will remove agglomerated stickies and pitch from the recycled fiber stock. Downstream of the mechanical separation stage, the stock contains few stickies. This will allow for less expensive processing as

other chemical treatments are not needed to eliminate the detrimental effects of the stickies and pitch deposits.

The oil soluble or water-dispersible hydrophobic polymers that are
5 useful in the present invention are selected primarily for their
hydrophobicity and, in some measure, their ability to provide tackiness to
stickies and pitch in the aqueous phase. Examples of these polymers
include, but are not limited to, phenol formaldehyde resin having a
molecular weight of about 300 to about 1750, preferably the molecular
10 weight is about 300, available as Cardolite NC-511 from Cardolite.

The hydrophobic polymers useful in this invention further include
but are not limited to mixtures of tackified styrene-isoprene-styrene and
styrene-butadiene based rubbers. These mixtures comprise by weight
15 from about 11 to about 28% styrene-isoprene-styrene rubber having a
molecular weight of about 135,000 and from about 11 to about 28%
styrene-butadiene rubber, and from about 58 to 68 percent resin
tackifiers. Typically the weight ratio of styrene-isoprene-styrene rubber to
styrene-butadiene rubber is less than or equal to 1. The total amount of
20 rubbers range from about 32 to 42 percent. The tackifiers comprise
hydrocarbon resins, such as a low molecular weight aliphatic
or aromatic hydrocarbon, a rosin-based resin tackifier, and a polyethylene
polyester resin. This mixture is available from Avery Dennison as Avery
Dennison AE-3610.

25

Other oil soluble or water-dispersible polymers useful in the
methods of the present invention include, but are not limited to, alkylated
vinyl pyrrolidone polymers having molecular weights from about 7300 to

about 8600 and are available from ISP as Ganex V-220 and Ganex V-216; polymethacrylate available as Acryloid 966 from Rohm & Haas; and hydroxy terminated 1, 3-butadiene homopolymer available from Elf Atochem as Polybd R-45HT resin; perfluoroalkyl polymers having
5 molecular weight from about 2300 to about 4300 available as Lodyne SP-101A and Lodyne SP-35A available from Ciba-Geigy.

The oil soluble or water-dispersible surfactants have HLB from about 1 to about 10. The surfactants useful in the present invention
10 include but are not limited to ethylene oxide (EO)-propylene oxide (PO) block copolymers having an HLB of about 1 and a molecular weight of about 3800 and an HLB of about 4 and a molecular weight of about 5000 available from BASF as Pluronic L-101 and Pluronic L-122 respectively;
15 C₁₂ to C₁₄ alcohol ethoxylate having an HLB of about 6 and C₁₂ to C₁₆ alcohol ethoxylate having an HLB of about 4.4, both available from Vista Chemical respectively as Alfonic 1214 GC-2 and Alfonic 1216-22; and C₁₁ to C₁₅ secondary alcohol ethoxylate having HLB of about 8 and available as Tergitol 15-S-3 from Union Carbide.

20 Other suitable surfactants include but are not limited to ethoxylated propoxylated fatty acids having HLB's from about 3 to about 4.5 and molecular weights of about 1300 to about 1400. This compound is available commercially as Pegol L31 Ditallate from Rhone-Poulenc; coconut diethanolamide having a molecular weight of about 200 to about
25 350 from Witco as Varamide A2; and alkyl phenol ethoxylate having an HLB of about 4.6 available as Igepal CO-210 from Rhone-Poulenc. The present inventor anticipates that alcohol ethoxylates having from about C₈ to about C₂₂ will also be effective in the methods of the present invention.

The oil soluble or water-dispersible resin tackifiers useful in the methods of the present invention include but are not limited to rosin based resin having a softening point of about 80 to about 86°C available as Zonester 85 and terpene based resins having a softening point of about 22 to about 118°C available as Zonarez ALPHA 25, Zonarez 7115 and Zonarez B-115 from Arizona Chemical.

The oil soluble or water-dispersible solvents that are useful in the present invention are aliphatic hydrocarbons which include but are not limited to 1-methyl-4-isopropenyl-1-cyclohexene having a molecular weight of about 135 and available from TR-Metro as d-limonene; aliphatic hydrocarbons having molecular weights of about 120 to about 180 available as 140 solvent from Ashland; toluene; decyl alcohol available as Epal 10 from Ethyl Corp.; n-methyl-2-pyrrolidone available as NMP from ARCO and triethylene glycol.

Other suitable solvents include but are not limited to polybutene having a molecular weight of about 350 to about 500 available from AMOCO as Indopol L-14; terpene dimer having a molecular weight of about 270 available as Acintene SDO-ACP from Arizona Chemical; and an epoxidized soybean oil having a molecular weight of about 1000 available from C. P. Hall Company as Plasthall ESO.

For purposes of the present invention, the phrase "at least two of" refers to a composition comprising a polymer and a solvent; a polymer and a surfactant; a polymer and a tackifier; a solvent and a surfactant; or a polymer, a tackifier, a solvent, and a surfactant; or any combinations

thereof. The terms polymer, solvent, tackifier and surfactant may include one or more of the designated class of compounds.

The compositions of the present invention are generally employed
5 in a weight ratio of 1:9 to 9:1. As such, when two components are present, their weight ratio will range from about 1:9 to 9:1. When three components are present, the weight ratios of one will range from about 1:9 to 9:1 to each of the other two components, and so on for four components when they are all present in the composition.

10

The compositions of the present invention may be added to the pulper or the stock chest in an amount ranging from about 5 parts per million parts oven dry (O.D.) fiber to about 10,000 parts per million parts O.D. fiber. Preferably, from about 50 to about 5,000 parts are added per
15 million part O.D. fiber. The effective amount of the composition to be added depends on a number of variables including the pH of the system, hardness of the water, temperature of the water; additional additives, consistency of furnish, and quantity and type of stickies and pitch in the furnish.

20

The compositions of the present invention may be compatible with other pulp and papermaking additives. These include starches, titanium dioxide, defoamers, wet strength resins, sizing aids and biocides.

25

The compositions of the present invention are effective at temperatures ranging from room temperature ($\approx 75^{\circ}\text{F}$) to about 180°F . These compositions are effective at a pH range of about 3 to about 12.

The mechanical separation of the agglomerated stickies and pitch follows the chemical treatment. This mechanical separation lies between the pulper or the stock storage chest and the headbox. These mechanical separation processes are performed with one or more of
5 primary, secondary and tertiary screens; forward, reverse, and throughrow cleaners; flotation devices; and centrifugal devices.

The use of slotted screens is preferred in the methods of the present invention. Typically, mills employ a fine screen of 0.006 inch (152
10 micron) to 0.012 inch (305 micron) slots. These finer diameter screens sometimes are problematic as their screening capacity sacrifices throughput. The method of the present invention allow mill personnel to employ a larger diameter screen to capture the agglomerated stickies and pitch particles without sacrificing throughput.

15

The data set forth below were developed to demonstrate the unexpected results occasioned by use of the present invention. The following examples are included as being illustrations of the invention and should not be construed as limiting the scope thereof.

Table I

	<u>Formulation</u>	<u>Component #1</u>	<u>Component #2</u>	<u>Component #3</u>
5				
	1	1-Methyl-4-isopropenyl-1-cyclohexene (MW=136)	alkylphenol ethoxylate avg. MW=280, HLB=4.6	EP-PO Block copolymer avg. MW=3800, HLB=1
10	2*	Aliphatic hydrocarbons MW=128 to 176	alkylphenol ethoxylate avg. MW=280, HLB=4.6	EO-PO block copolymer avg. MW=3800, HLB=1
	3	Aliphatic hydrocarbons MW=128 to 176	rosin acid esters softening point 82°C	EO-PO block copolymer avg. MW=5000, HLB=4
15	4	1-methyl-4-isopropenyl-1-cyclohexene (MW=136)	rosin acid esters softening point 82°C	EO-PO block copolymer avg. MW=5000, HLB=4
	5	polybutene, MW=350 to 500	polymethacrylate spec. grav. =0.90 to 0.92	-
20	6	terpene dimer MW=270	polymethacrylate spec. grav. =0.90 to 0.92	-
	7	polybutene, MW=350 to 500	phenol formaldehyde resin avg. MW=302	-
25	8	C ₁₀ alcohol, MW=158	phenol formaldehyde resin avg. MW=302	-
30	9	ethoxylated propoxylated fatty acid	phenol formaldehyde resin avg. MW=302	-
	10	terpene dimer MW=270	phenol formaldehyde resin resin, avg. MW=302	-
35	11	polymethacrylate spec. grav. =0.90 to 0.92	phenol formaldehyde resin avg. MW=302	-
	12	polybutene, MW=350 to 500	alkylated vinylpyrrolidone polymer (avg. MW=8600)	-
40	13	phenol formaldehyde resin avg. MW=302	alkylated vinylpyrrolidone polymer, avg. MW=7300	-
45	14	terpene dimer MW=270	coconut diethanolamide MW = 204 to 344	-
50	15	alkylphenol ethoxylate avg. MW=280, HLB 4.6	EO-PO block copolymer avg. MW=3800, HLB=1	aliphatic hydrocarbons MW=128 to 176

Table I (continued)

	<u>Formulation</u>	<u>Component #1</u>	<u>Component #2</u>	<u>Component #3</u>
5	16	alkylphenol ethoxylate avg. MW=280, HLB=4.6	branched alcohol ethoxylate, avg. MW=332, HLB=8	triethylene glycol
10	17	mixture of S-I-S and S-B copolymers	aromatic hydrocarbon avg. MW=92	-
	18	mixture of S-I-S and S-B copolymers	linear alcohol C ₁₂ - C ₁₄ ethoxylate, HLB=6	aromatic hydrocarbon avg. MW=92
15	19	mixture of S-I-S and S-B copolymers	linear alcohol C ₁₂ - C ₁₄ ethoxylate, HLB=6	1-methyl-4-isopropenyl -1-cyclohexene, MW=136
	20	mixture of S-I-S and S-B copolymers	alkylphenol ethoxylate avg. MW=280, HLB=4.6	1-methyl-4-isopropenyl- 1-cyclohexene, MW=136
20	21	mixture of S-I-S and S-B copolymers	linear alcohol C ₁₂ - C ₁₆ ethoxylate, HLB=4.4	1-methyl-4-isopropenyl- 1-cyclohexene, MW=136
25	22	polyterpene (softening point 115°C)	linear alcohol C ₁₂ -C ₁₄ ethoxylate HLB=6	1-methyl-4-isopropenyl- 1-cyclohexene, MW=136
	23	mixture of S-I-S and S-B copolymers	linear alcohol C ₁₂ -C ₁₄ ethoxylate HLB=6	n-methyl-2-pyrrolidone, NW=99
30	24	polyterpene (softening point 25°C)	1-methyl-4-isopropenyl- 1-cyclohexene, NW=136	

*Also contains perfluoroalkyl polymer, MW=4236

S-I-S is styrene-isoprene-styrene

35 S-B is styrene-butadiene

Laboratory Pulper Agglomeration Test

40 An unbleached kraft paper, cut into 1" x 2" pieces was repulped to
5% consistency at 140°F (~60°C) and neutral pH for 60 minutes using a
Formax Hydrapulper, to make a clean, uncontaminated furnish. The
pressure-sensitive adhesive contaminated furnish was prepared using the
same procedure, except an adhesive tape (3M packaging tape, model
45 No. 3750-G) was attached to a portion of the unbleached kraft paper. For

hot melt and wax contaminants, these were added as solid particles directly to the clean fiber immediately following repulping.

A laboratory pulper was then used to evaluate the agglomeration tendency of stickies using the previously repulped furnish. For the studies involving the pressure-sensitive adhesive, the adhesive contaminated furnish was employed, otherwise the clean furnish was used. The repulped furnish was placed in the laboratory pulper and diluted to 4% consistency at neutral pH. The furnish was heated to the desired temperature and the shear force applied at 1000 rpm. The contaminants, 0.714% wt. based on O.D. fiber, were added to the vortex of the furnish and allowed to mix for approximately one minute. The average size of hot melt and wax contaminants that were added to the pulper was about 200 microns. The furnish containing the test contaminants was then treated with an agglomeration agent at the dosage of 2 - 20 pounds per ton based on O.D. fiber. After 30 minutes treatment, the furnish was run across a 0.006 inch flat screen to collect the stickies agglomerates. The agglomerates were examined for particle size distribution using a Cue-2 image analyzer.

Table II

Particle Size Distribution of Agglomerates for Hot Melt and Pressure-Sensitive Adhesive				
Treatment	Particle Size Distribution of Hot Melt Agglomerates* (microns)		Particle Size Distribution of Pressure-Sensitive Adhesive Agglomerates* (microns)	
	@ 10#/T	@ 20 #/T	@ 10#/T	@ 20 #/T
Untreated	434 ~ 1570		248 ~ 536	
Formulation #1	958 ~ 3.548	1.932 ~ 4.028	385 ~ 1.044	627 ~ 1.336
Formulation #2	970 ~ 3.180	1.526 ~ 3.290	484 ~ 1.201	770 ~ 1.540
Formulation #3	--	770 ~ 1.540	--	--
Formulation #4	--	1.134 ~ 2.150	--	--
Formulation #5	916 ~ 3.860	862 ~ 3.240	--	305 ~ 525

Table II-(continued)

Particle Size Distribution of Agglomerates for Hot Melt and Pressure-Sensitive Adhesive					
Treatment		Particle Size Distribution of Hot Melt Agglomerates* (microns)		Particle Size Distribution of Pressure-Sensitive Adhesive Agglomerates* (microns)	
5	Formulation #6	534 ~ 1.486	1.106 ~ 1.992	--	225 ~ 370
10	Formulation #7	778 ~ 2.658	1.322 ~ 2.254	289 ~ 1.029	494 ~ 1.275
	Formulation #8	996 ~ 3.442	1.196 ~ 2.178	377 ~ 546	242 ~ 803
	Formulation #9	1.084 ~ 3.500	576 ~ 2.930	371 ~ 848	323 ~ 1.087
	Formulation #10	890 ~ 2.610	994 ~ 2.732	351 ~ 712	237 ~ 1.345
	Formulation #11	--	1.256 ~ 2.616	--	--
15	Formulation #12	537 ~ 2.483	638 ~ 1.983	357 ~ 1.000	395 ~ 742
	Formulation #13	656 ~ 2.231	972 ~ 2.552	282 ~ 803	321 ~ 593
	Formulation #14	763 ~ 3.099	524 ~ 2.405	385 ~ 1.235	314 ~ 1.458
	Formulation #15	674 ~ 2.784	1.864 ~ 3.220	466 ~ 841	554 ~ 1.110
	Formulation #16	1.218 ~ 3.540	1.204 ~ 4.062	331 ~ 897	717 ~ 1.650
20	Formulation #17	874 ~ 2362	1056 ~ 2649	375 ~ 754	519 ~ 962
	Formulation #18	604 ~ 1812	1267 ~ 2869	386 ~ 926	645 ~ 1067
		82 ~ 187 (@ 110°F)		56 ~ 287 (@ 110°F)	
	Untreated	94 ~ 275 (@ 130°F)		55 ~ 336 (@ 130°F)	
	Formulation #19	212 ~ 434 (@ 110°F)		127 ~ 345 (@ 110°F)	
25	(10 #/T)	200 ~ 394 (@ 130°F)		116 ~ 337 (@ 130°F)	
	Formulation # 20				
	(10 #/T)	131 ~ 413 (@ 130°F)		264 ~ 332 (@ 130°F)	
	Formulation #21				
	(10 #/T)	419 ~ 926 (@ 130°F)		329 ~ 464 (@ 130°F)	
30	Formulation #22				
	(10 #/T)	305 ~ 768 (@ 110°F)		165 ~ 407 (@ 110°F)	
	Formulation #23				
	(10 #/T)	245 ~ 546 (@ 110°F)		149 ~ 348 (@ 110°F)	
	Formulation #24				
35	(10 #/T)	178 ~ 442 (@ 110°F)		163 ~ 314 (@ 110°F)	

*Test Conditions: Temp.: 150 F pH 7~ 8 Time: 30 min. Shear: 1000 rpm

Due to the variable nature of stickies, their melting points,
softening points, glass transition temperatures, tackiness and
hydrophobicity will differ. As such, the repulping temperature, pH, time,

shear force and chemical treatment all have an impact on stickies agglomeration.

- As demonstrated in Table II, few noticeable hot melt agglomerates were observed when the temperature was below 140°F without chemical treatments. Formulations #1 to 18 enhanced the hot melt agglomeration only at the temperatures of 150°F. In general, the particle size distribution of hot melt agglomerates shifted to larger sizes as the dosage of treatment increased from 10 lbs. per ton to 20 lbs. per ton.
- Formulations 19 to 24 were very effective at agglomerating hot melts even when the temperature was as low as 110°F.

Table III

Particle Size Distribution of Agglomerates for Mixed Stickies

	Treatment (10 #/T)	Particle Size Distribution of Hot Melt & Wax Agglomerates	Particle Size Distribution of Pressure-Sensitive Adhesive & Wax Agglomerates Temp. @
		Temp. @ 140 °F (microns)	130 °F (microns)
20	Untreated	325 ~ 1.005	263 ~ 590
	Formulation #1	385 ~ 1.771	851 ~ 1.390
	Formulation #2	581 ~ 1.151	723 ~ 926
	Formulation #5	342 ~ 1.172	-
	Formulation #6	571 ~ 1.290	-
25	Formulation #7	688 ~ 1.153	545 ~ 1.096
	Formulation #8	596 ~ 1.250	674 ~ 928
	Formulation #9	543 ~ 1.573	646 ~ 1.069
	Formulation #10538	~ 1.835	606 ~ 1.048
	Formulation #12536	~ 1.111	629 ~ 982
30	Formulation #13669	~ 822	801 ~ 1.082
	Formulation #14865	~ 1.292	816 ~ 1.356
	Formulation #15	815 ~ 1.646	848 ~ 1.071
	Formulation #16487	~ 963	629 ~ 1.638
	Untreated (130°F)	180 ~ 939 (130°F)	60 ~ 352
35	Formulation #19 (130°F)	622 ~ 1.599(130°F)	248 ~ 586
	Formulation #20 (130°F)	497 ~ 1.365(130°F)	314 ~ 607
	Formulation #21 (130°F)	742 ~ 1.997(130°F)	372 ~ 658
40			

As demonstrated in Table III, for formulations 1 to 16, hot melt-wax agglomeration was observed when the mixed stickies were treated at 140°F or above. The increase in the minimal size of hot melt-wax agglomerates should improve the screen's removal efficiency. At a neutral pH, 130°F and 10 pounds per ton of treatment, formulations #19 and 20 performed equally well and were more effective than formulations #1 to 16. A similar trend was observed for the pressure-sensitive adhesive-wax agglomeration.

Table IV

Particle Size Distribution of Sticky Agglomerates Derived from Hot Melt, Pressure-Sensitive Adhesive and Wax

	Treatment (10#/T)	Particle Size Distribution Temp. @130°F (microns)	Particle Size Distribution Temp. @140°F (microns)	Particle Size Distribution Temp. @150°F (microns)
15	Untreated	255 ~ 685	312 ~ 674	375 ~ 1,486
	Formulation #1	647 ~ 920	639 ~ 1,156	805 ~ 2,907
	Formulation #2	381 ~ 1,005	397 ~ 1,057	654 ~ 3,343
20	Formulation #5	457 ~ 718	470 ~ 1,780	616 ~ 1,840
	Formulation #6	—	361 ~ 811	481 ~ 2,440
	Formulation #7	419 ~ 803	535 ~ 918	617 ~ 1,747
	Formulation #8	—	369 ~ 934	516 ~ 1,351
	Formulation #9	708 ~ 948	557 ~ 939	402 ~ 2,194
25	Formulation #10	631 ~ 875	388 ~ 1,675	772 ~ 1,845
	Formulation #12	371 ~ 679	320 ~ 569	692 ~ 2,673
	Formulation #13	530 ~ 812	592 ~ 756	433 ~ 2,263
	Formulation #14	400 ~ 810	609 ~ 1,123	871 ~ 2,577
	Formulation #15	416 ~ 959	408 ~ 1,163	812 ~ 2,017
30	Formulation #16	297 ~ 759	601 ~ 956	454 ~ 1,528
	Formulation #21	—	346 ~ 1,504	—

Test Conditions: pH: 7 ~ 8 Shear: 1000 rpm Consistency: 4% Time: 30 minutes

Table IV demonstrates that mixed stickies of hot melt/pressure-sensitive adhesive/wax can be agglomerated at 130°F or above when they are treated with agglomeration agents. The average agglomerate size increased with increasing temperature.

Table V

Particle Size Distribution of Mixed Stickies Agglomerates
When Treated with Formulation #21 at Various Dosages

Treatment	Particle Size Distribution of Sticky Agglomerates (microns)		
	Temp. @ 125°F & pH 11	Temp. @ 140°F & pH 7	Temp. @ 125°F & pH 11
Untreated	89 ~ 700	130 ~ 715	254 ~ 766
Formulation #21 @ 2 #/T	317 ~ 943	230 ~ 795	350 ~ 1,082
Formulation #21 @ 5 #/T	259 ~ 815	268 ~ 1,179	314 ~ 1,164
Formulation #21 @ 10 #/T	332 ~ 934	346 ~ 1,504	446 ~ 1,836

As demonstrated in Table V, Formulation #21 proved very effective at agglomerating mixed stickies at pH 11 and a temperature of 125°F. This formulation was even more efficacious at temperatures of 140°F. Stickies agglomeration tendency was enhanced at a higher pH with the treatment.

A laboratory study was conducted to evaluate the efficiency of stickies removal by a flat screen using the chemical agglomeration mechanism. The experimental procedure involved dosing laboratory pulpers with a known quantity of stickies, including hot melt, pressure-sensitive adhesive, and wax, and performing the pulper agglomeration test for control and treated runs. The rejects from screening (0.006 inch slot) were collected, dried, and accurately weighed. The percent rejects level (i.e., % removal) could then be determined.

Table VI**Stickies Removal by a Flat Screen with the Chemical Agglomeration****Mechanism**

5	Dosage of			
	<u>Formulation #21</u>	<u>Temperature (°F)</u>	<u>pH</u>	<u>% Stickies Removal</u>
	0 lbs/T	125	11	67.2
	2 lbs/T	125	11	100.0
	5 lbs/T	125	11	100.0
10	10 lbs/T	125	11	100.0
	0 lbs/T	140	7	47.4
	2 lbs/T	140	7	80.7
	5 lbs/T	140	7	100.0
15	10 lbs/T	140	7	78.0
	0 lbs/T	140	11	65.9
	2 lbs/T	140	11	100.0
	5 lbs/T	140	11	82.7
20	10 lbs/T	140	11	100.0

Note: Stickies consisted of hot melt, pressure-sensitive adhesive, and wax.

As demonstrated on Table VI, due to stickies agglomeration, the
 25 percent of stickies removal with the treatment was consistently higher than
 those of the control runs. Under the tested conditions, an increase of 20
 to 30% removal was always obtained. This indicates that with the
 chemical treatment the stickies can be effectively removed by increasing
 their sizes with the aid of mechanical separation. By doing this, stickies
 30 can be removed from the papermaking system at an early stage, and will
 not cause problems downstream.

While this invention has been described with respect to particular
 embodiments thereof, it is apparent that numerous other forms and
 35 modifications of this invention will be obvious to those skilled in the art.

The appended claims and this invention generally should be construed to cover all such obvious forms and modifications which are within the true spirit and scope of the present invention.

Having thus described the invention, what I claim is:

1. A method for removing stickies and pitch from papermaking fiber in a pulp and papermaking system comprising the steps of adding to said fiber in the pulper or storage chest a composition comprising at least two of an oil soluble or water-dispersible, hydrophobic polymer, an oil soluble or water-dispersible surfactant having an HLB of about 1 to about 10, a tackifier, and an oil soluble or water-dispersible solvent wherein said composition will cause the agglomeration of stickies and pitch; and subsequently mechanically separating said agglomerated stickies and pitch from said fiber furnish.

2. The method as claimed in claim 1 wherein said hydrophobic polymer is selected from the group consisting of mixtures of tackified styrene-isoprene-styrene and styrene-butadiene based rubbers, phenol-formaldehyde resins, alkylated vinyl pyrrolidones, polymethacrylates, perfluoroalkyl polymer, and hydroxy terminated 1, 3-butadiene homopolymer.

3. The method as claimed in claim 2 wherein said mixtures of tackified styrene-isoprene-styrene and styrene-butadiene based rubbers comprise by weight from about 11 to about 28% styrene-butadiene rubber and from about 11 to about 28% styrene-isoprene-styrene rubber.

4. The method as claimed in claim 3 wherein the weight ratio of said styrene-isoprene-styrene rubber to said styrene-butadiene rubber is less than or equal to 1.

5. The method as claimed in claim 3 wherein said mixtures of tackified styrene-isoprene-styrene and styrene-butadiene further comprise a hydrocarbon and rosin-based resin tackifiers.

6. The method as claimed in claim 2 wherein said phenol-formaldehyde resins have a molecular weight of about 300 to about 1750.

7. The method as claimed in claim 6 wherein said phenol-formaldehyde resins have a molecular weight of about 300.

8. The method as claimed in claim 2 wherein said alkylated vinyl pyrrolidones have a molecular weight of about 7300 to about 8600.

9. The method as claimed in claim 2 wherein said neat liquid polymethacrylates have a specific gravity of about 0.90 to about 0.92.

10. The method as claimed in claim 2 wherein said perfluoroalkyl polymers have a molecular weight of about 2300 to about 4300.

11. The method as claimed in claim 2 wherein said tackifier is selected from the group consisting of rosin based resins and terpene based resins.

12. The method as claimed in claim 11 wherein said resins have a softening point from about 20°C to about 120°C.

13. The method as claimed in claim 1 wherein said surfactant is selected from the group consisting of an ethylene oxide-propylene oxide

block copolymer, alcohol ethoxylate, ethoxylated propoxylated fatty acid, coconut diethanolamide, and alkyl phenol ethoxylate.

14. The method as claimed in claim 13 wherein said surfactant is
5 selected from the group consisting of an ethylene oxide-propylene oxide
block copolymer having an HLB of about 1 and a molecular weight of
about 3800, and an HLB of about 4 and a molecular weight of about 5000,
a C₁₂ to C₁₄ alcohol ethoxylate having an HLB of about 6, a C₁₂ to C₁₆
alcohol ethoxylate having an HLB of about 4.4, a C₁₁ to C₁₅ secondary
10 alcohol ethoxylate having an HLB of about 8, an ethylene oxide propylene
oxide fatty acid having an HLB of about 3 to about 4.5 and a molecular
weight of about 1300 to about 1400, a coconut diethanolamide having a
molecular weight of about 200 to about 350, and an alkyl phenol ethoxylate
having an HLB of about 4.6.

15. The method as claimed in claim 1 wherein said solvent is
selected from the group consisting of 1-methyl-4-isopropenyl-1-
cyclohexene, aliphatic hydrocarbons, toluene, alcohols, triethylene glycol,
polybutene, terpene dimer, n-methyl-2-pyrrolidone and epoxidized
soybean oil.

16. The method as claimed in claim 15 wherein said solvent is
selected from the group consisting of aliphatic hydrocarbons having
molecular weights from about 120 to about 180, a polybutene having a
molecular weight of about 350 to about 500, a terpene dimer having a
molecular weight of about 270, and an epoxidized soybean oil having a
molecular weight of about 1000.

17. The method as claimed in claim 1 wherein said composition is added to said pulper or stock chest in an amount ranging from about 5 parts to about 10,000 parts per million parts O.D. fiber.

18. The method as claimed in claim 1 wherein said pulp and papermaking system has a pH of about 3 to about 12.

19. The method as claimed in claim 1 wherein said pulp and papermaking system is at a temperature of about 50°F to about 180°F.

20. The method as claimed in claim 1 wherein said mechanical separation is performed with a device selected from the group consisting of primary, secondary, and tertiary screens; forward, reverse, and throughrow cleaners; flotation devices; and centrifugal devices.

21. A stickies and pitch agglomeration composition comprising at least two of an oil soluble or water-dispersible, hydrophobic polymer, an oil soluble or water-dispersible surfactant having an HLB of about 1 to about 10, a tackifier, and an oil soluble or water-dispersible solvent.

22. The composition as claimed in claim 21 wherein said hydrophobic polymer is selected from the group consisting of mixtures of tackified styrene-isoprene-styrene and styrene-butadiene based rubbers, phenol-formaldehyde resins, alkylated vinyl pyrrolidones,
5 polymethacrylates, perfluoroalkyl polymer, and hydroxy terminated 1,3-butadiene homopolymer.

23. The composition as claimed in claim 22 wherein said mixtures of tackified styrene-isoprene-styrene and styrene-butadiene based rubbers comprise by weight from about 11 to about 28% styrene-butadiene rubber and from about 11 to about 28% styrene-isoprene-styrene rubber.

24. The composition as claimed in claim 23 wherein the weight ratio of said styrene-isoprene-styrene rubber to said styrene-butadiene rubber is less than or equal to 1.

25. The composition as claimed in claim 23 wherein said mixtures of tackified styrene-isoprene-styrene and styrene-butadiene further comprise a hydrocarbon and rosin-based resin tackifiers.

26. The composition as claimed in claim 22 wherein said phenol-formaldehyde resins have a molecular weight of about 300 to about 1750.

27. The composition as claimed in claim 26 wherein said phenol-formaldehyde resins have a molecular weight of about 300.

28. The composition as claimed in claim 22 wherein said alkylated vinyl pyrrolidones have a molecular weight of about 7300 to about 8600.

29. The composition as claimed in claim 22 wherein said neat liquid polymethacrylates have a specific gravity of about 0.90 to about 0.92.

30. The composition as claimed in claim 22 wherein said perfluoroalkyl polymers have a molecular weight of about 2300 to about 4300.

31. The composition as claimed in claim 21 wherein said tackifier is selected from the group consisting of rosin based resins and terpene based resins.

32. The composition as claimed in claim 31 wherein said resins have a softening point from about 20°C to about 120°C.

33. The composition as claimed in claim 21 wherein said surfactant is selected from the group consisting of an ethylene oxide-propylene oxide block copolymer, alcohol ethoxylate, ethoxylated propoxylated fatty acid, coconut diethanolamide, and alkyl phenol ethoxylate.

34. The composition as claimed in claim 33 wherein said surfactant is selected from the group consisting of an ethylene oxide-propylene oxide block copolymer having an HLB of about 1 and a molecular weight of about 3800, and an HLB of about 4 and a molecular weight of about 5000, a C₁₂ to C₁₄ alcohol ethoxylate having an HLB of about 6, a C₁₂ to C₁₈ alcohol ethoxylate having an HLB of about 4.4, a C₁₁ to C₁₅ secondary alcohol ethoxylate having an HLB of about 8, an ethylene oxide propylene oxide fatty acid having an HLB of about 3 to about 4.5 and a molecular weight of about 1300 to about 1400, a coconut diethanolamide having a molecular weight of about 200 to about 350, and an alkyl phenol ethoxylate having an HLB of about 4.6.

35. The composition as claimed in claim 21 wherein said solvent is selected from the group consisting of 1-methyl-4-isopropenyl-1-cyclohexene, aliphatic hydrocarbons, toluene, alcohols, triethylene glycol, polybutene, terpene dimer, n-methyl-2-pyrrolidone and epoxidized soybean oil.

36. The composition as claimed in claim 35 wherein said solvent is selected from the group consisting of aliphatic hydrocarbons having molecular weights from about 120 to about 180, a polybutene having a molecular weight of about 350 to about 500, a terpene dimer having a molecular weight of about 270, and an epoxidized soybean oil having a molecular weight of about 1000.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US98/05261

A. CLASSIFICATION OF SUBJECT MATTER

IPC(6) : Please See Extra Sheet

US CL : 162/165, 168.1, 168.5, 168.7; 524/505, 509, 516, 524, 525

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 162/165, 168.1, 168.5, 168.7; 524/505, 509, 516, 524, 525

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

NONE

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

APS

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 5,618,861 A (HLIVKA et al) 08 April 1997, columns 3-5.	1-36
X	US 5,415,739 A (FURMAN, Jr. et al) 16 May 1995, columns 2-4.	1-36
X	US 5,368,694 A (ROHLF et al) 29 November 1994, columns 4-6.	1-36
X	US 5,300,194 A (WELKENER et al) 05 April 1994, columns 2-5.	1-36
X	US 4,643,800 A (MALONEY et al) 17 February 1987, columns 3-7.	1-36

☐ Further documents are listed in the continuation of Box C.☐ See patent family annex.

* Special categories of cited documents:	*T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
A document defining the general state of the art which is not considered to be of particular relevance	*X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
E earlier document published on or after the international filing date	*Y* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
L document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	*A* document member of the same patent family
O document referring to an oral disclosure, use, exhibition or other means	
P document published prior to the international filing date but later than the priority date claimed	

Date of the actual completion of the international search

15 JUNE 1998

Date of mailing of the international search report

08 JUL 1998

Name and mailing address of the ISA/US
Commissioner of Patents and Trademarks
Box PCT
Washington, D.C. 20231

Facsimile No. (703) 305-3230

Authorized officer

PETER D. MULCAHY

Telephone No. (703) 308-2351

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US98/05261

A. CLASSIFICATION OF SUBJECT MATTER:

IPC (6):

D21H 11/00, 13/00, 15/00, 17/00, 19/00, 21/00, 23/00, 25/00, 27/00; CO8L 53/00, 61/04, 39/00, 31/04, 27/00